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**MATERIALS AND PROCESS CHARACTERIZATION TO
DETERMINE THE EFFECT OF HIGH INTENSITY
INFRARED LASER RADIATION ON
A COMPOSITE MATERIAL**

JANET S. PERKINS et al.
COMPOSITES DEVELOPMENT DIVISION

March 1985

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
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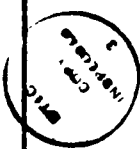
ABSTRACT

Measurements were made of the true (black body) surface temperature of a well-characterized metal-doped carbon-carbon composite during irradiation with an intense, continuous-wave, CO₂-laser beam along with spectrometric determination of the temporal variation of C₂ⁿ and C₃ⁿ species appearing in the laser-induced plume in a high vacuum environment. Various analytical methods used for post-burn analyses, during irradiation analyses, and material characterization are detailed. Thermodynamic aspects are considered. A synopsis of the stages in laser-beam/composite interaction derived from this study is included.

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**MATERIALS AND PROCESS CHARACTERIZATION TO DETERMINE THE
EFFECT OF HIGH INTENSITY INFRARED LASER RADIATION ON A
COMPOSITE MATERIAL**

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INTRODUCTION

A wide variety of characterization methods have been used to determine the effect of irradiating a specific composite material with high intensity infrared laser radiation. The material, being a metal-doped carbon/carbon composite, was intractable to the usual methods of analysis; the behavior under intense radiation was unexpected and, at the time, unexplained; and the desirability of computer modeling required knowledge of the principal mechanisms of radiant energy absorption and redistribution.

This study of the effect of an intense infrared laser beam on a high-temperature, metalated ceramic system became feasible by use of relatively new and powerful analytical techniques.

The examination we performed can be divided into three areas of concentration, these being Materials Characterization, Laser Testing, and Post-burn Analyses. Study of these was correlated with data obtained from compilations, reports of experiments in the recent literature, and information privately communicated.

An outline of the principal stages in the laser beam/material interaction disclosed by this study is included.

MATERIALS CHARACTERIZATION

Initial studies on the effect of laser beam interaction with a metal-doped carbon/carbon composite showed that laser-produced front and back face temperature measurements during irradiation were reproducible providing material from a single sample source was used but that substantial variation in the resistance of the samples to damage occurred as different lots of material were tested. It thus became obvious that only by characterization of the different test samples would it be possible to unravel the effect of an intense laser beam on this type of material.

The material was a laminate of carbon cloth layers preimpregnated with a metal-containing resin. Different samples contained three to as many as a hundred and forty layers. These were successively subjected to curing, carbonizing, and graphitizing cycles under pressure, a process which allowed for substantial variation in the final products. These composite samples were black, opaque, and insoluble in most solvents, and analyses of the constituents required the development or refinement of special analytical methods. Especially useful techniques proved to be:

X-Ray diffraction measurements that showed carbon cloth/resin interaction during fabrication to produce varying amounts of the tungsten carbide products that consolidate the samples, i.e., WC, W_2C , and $\beta-WC_{1-x}$. These were produced at high temperatures by removal of the hydrogen, oxygen, nitrogen, and some of the carbon from the initial carbon, hydrogen, nitrogen, oxygen, and tungsten containing resin (Figure 1).

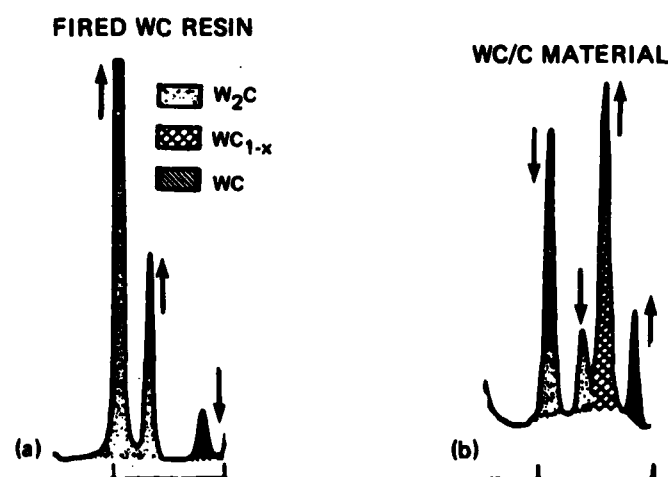


Fig. 1. X-Ray diffraction patterns. (a) Resin fired to 2800°C showing initial WC converted to W_2C ; (b) resin on carbon cloth similarly treated showing interaction of W_2C with cloth to produce $\beta-WC_{1-x}$ and WC in final composite, WC/C.

Elemental analyses that showed substantial deviations from the nominal W-content in a series of samples (Table 1) as well as uneven distribution of the metallic component throughout an individual sample.

Table 1. Variations in composition of eight nominally identical samples of 3-ply WC/C containing an added ingredient.

	C	W	X
11-1	86.1	8.7	4.4
11-2	83.6	9.1	4.9
11-3	85.1	9.3	5.0
11-4	83.4	10.1	4.8
11-5	86.4	8.7	4.5
11-6	76.7	12.7	6.0
11-7	76.5	12.5	5.0
11-8	80.2	6.8	5.5

X-Ray radiographs that also showed variations in the distribution of W in successive layers, the metal being concentrated near the top and bottom surfaces in some cases (Figure 2).



Fig. 2. X-Ray radiograph of 140-layer sample of WC/C showing concentration of WC's near top and bottom surfaces.

X-Ray fluorescence measurements that verified an uneven distribution of tungsten on the two surfaces, top and bottom, of a multilayered sample.

Apparent density measurements comparing samples cut from the surface and from the core.

Metallographic mounting of a sample, which otherwise appeared nearly homogeneous, that revealed the known layered structure. It also revealed another phase that either interpenetrated the layers or was spread across several adjacent layers during extended polish of the surface (Figure 3).

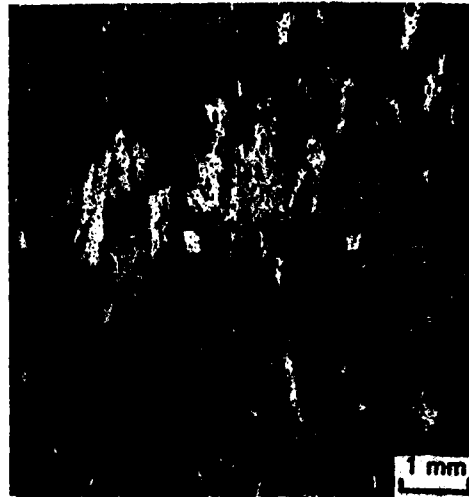


Fig. 3. Metallograph of a mounted sample showing 0°/90° cloth lay-up and a second phase (white).

LASER TEST DATA

During radiation with an intense continuous-wave (cw) CO₂-laser beam of 10.6- μ m wave length (a welding type laser), changes in the sample were monitored with:

Thermocouples which were buried at different depths under the area to be irradiated;

Optical pyrometers, Si (0.9 μ m), Ge (1.45 μ m), and/or Barnes (4.6 to 7.4 μ m) focused on the front and/or back face of the sample in the burn area;

A circularly variable filter for the infrared region monitoring a small area of the front face from which true front face temperatures could be derived by subsequent computer-matching with temperature-derived black body curves (Figures 4 and 5);

Video scans as well as high-speed cinematography, the former in real time, the latter at 100, 200, or 400 frames per second, to provide simultaneous records of each burn as viewed from the front, side, and, in some cases, the back;

Spectroscopy over the near UV to near IR region (200 to 1200 nm) either using an integrating monochrometer, a drum camera, or an optical multichannel analyzer (OMA), the last triggered to give eight instantaneous scans separated by selected equal time intervals during the course of the laser runs which varied from three to ten seconds in duration. These made possible the identification of molecular or atomic species in the plume, both adjacent and at a

nominal distance from the irradiated surface, and, with the last two instruments, over a temporal period after initiation of laser radiation. A typical scan (Figure 6) shows the emission spectra of C_2 and C_3 molecules directly in front of the burn surface as viewed from the side.

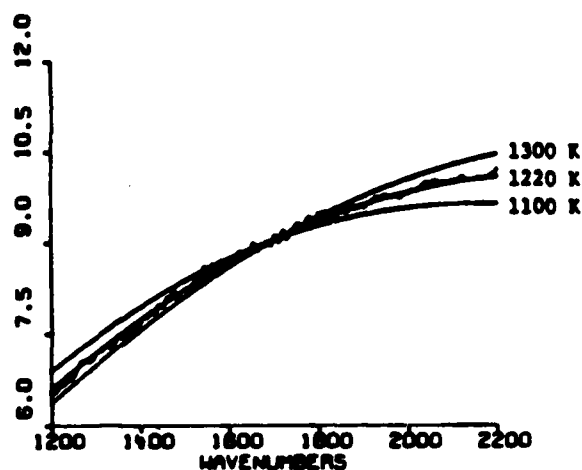


Fig. 4. Plots of corrected experimental test-case data compared with "best-fit" Planck function and $\pm 100K$ Planck functions. (OptiMetrics, Inc.)

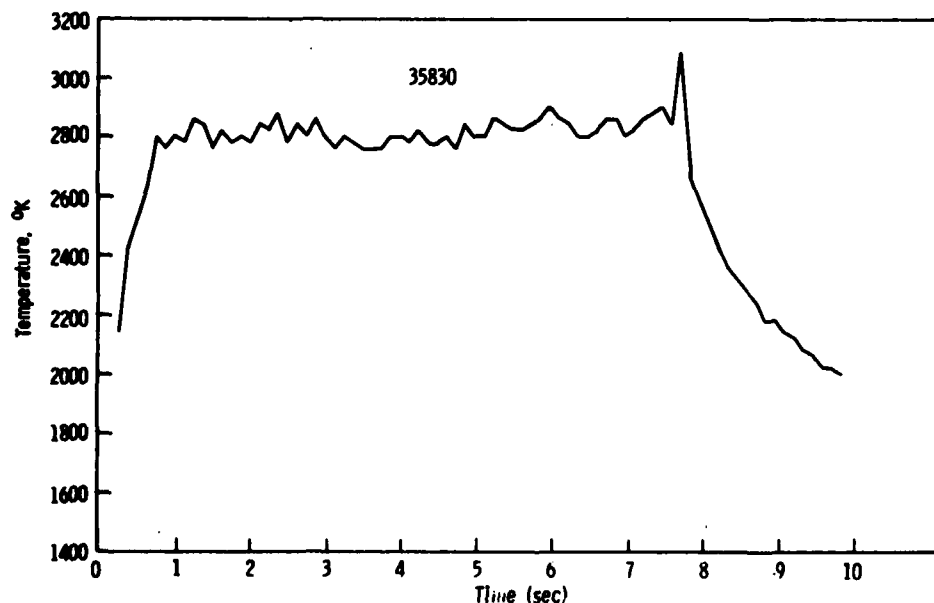


Fig. 5. True front-face temperatures of WC/C composite during irradiation with a 10.6- μm CO_2 -laser in a 77- μm vacuum chamber. These were derived by Planck function fitting of data measured using a circularly variable filter.

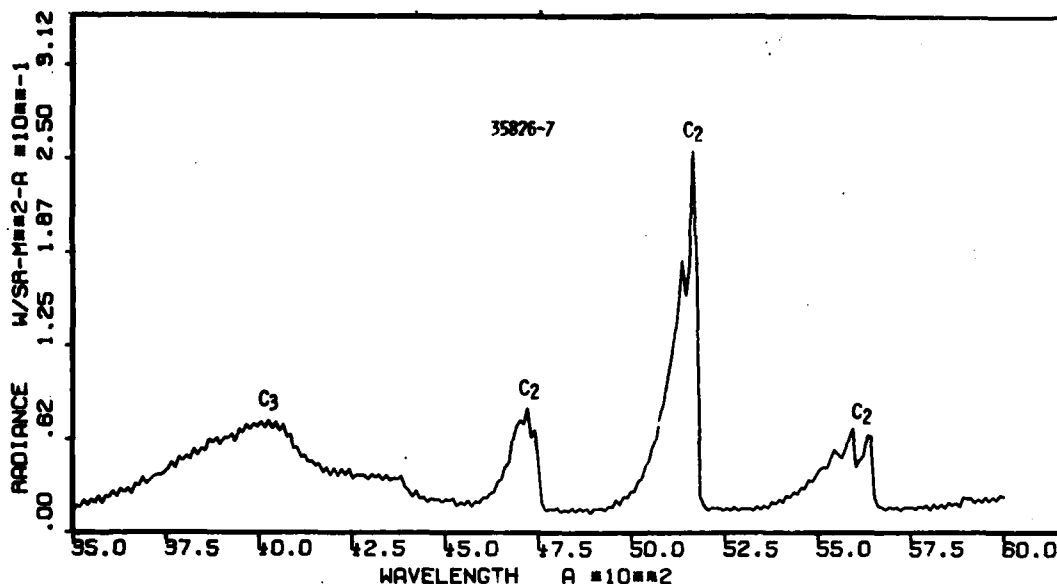


Fig. 6. OMA scan of the C₂ Swann bands and the nearly continuous C₃ emission bands seen from the side just in front of the WC/C surface during laser irradiation.

A visicorder trace of concurrent events tied all data together, i.e., the front, intermediate, and back face temperatures monitored by pyrometers and thermocouples, the triggering of OMA signals, the "on and off" timing of the laser, and any other measureable signal such as the rise and fall of pressure during laser runs in vacuum.

These records permitted review of the "in-situ" laser beam-sample interaction during post-radiation analysis of each laser run. They provided revealing temporal evidence of physical and chemical changes produced by the onset and course of steady laser irradiation of the sample. In addition, they allowed a study of continued changes during the immediate post-radiation period involving relaxation and redistribution of the absorbed electromagnetic energy.

We conclude that during irradiation the following stages are the most significant:

Selective photo excitation, predominantly of the carbon fabric by that portion of the beam absorbed and not reflected;

Multiphoton absorption of the low-energy 10.6- μ m photon (3 kcal/mole) to activate rotational, vibrational, and electronic transitions in carbon which produce incandescence and, when the radiation is intense or sufficiently prolonged, sublimation of the carbon;

Melting of the tungsten carbides in the matrix by the incandescent carbon cloth with release of postulated carbon atoms (C_1) from WC, forming tungsten-rich W_2C which then reacts with carbon in the matrix and cloth portions of the composite producing more molten WC that continues to release carbon.

This is a highly endothermic, cyclic process that causes the cloth to disappear (vaporize) if laser energy is sufficiently intense.

As the layer of liquid tungsten carbides becomes thicker, due to the disappearance of the carbon substrate, the endothermic reaction proceeds more rapidly. Simultaneously, the molten carbide surface layer generally becomes smoother and reflects more of the incident laser beam. These can counteract the accelerating effect and the reaction slows down to a steady state.

The most significant aspect of this mechanism, we believe, is the highly endothermic removal of monoatomic carbon (C_1) by the following mechanism:

At the liquid/vapor surface:



At the liquid/cloth surface:



which is equivalent to an energy absorption of 60 kJ/g of carbon released.

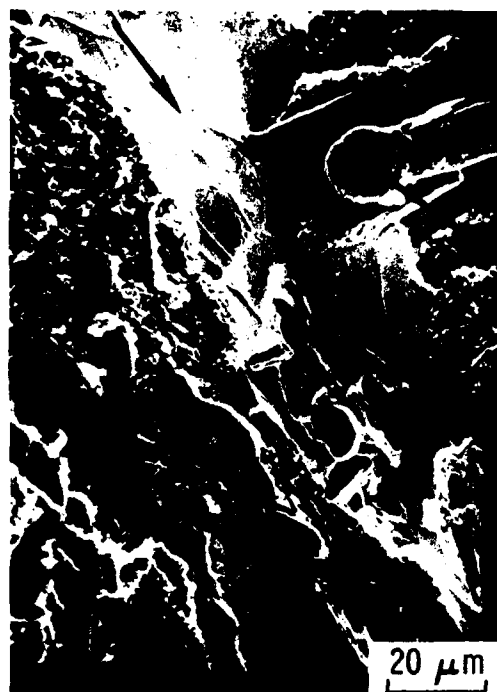
POST-BURN ANALYSES

Post-burn analyses strengthened our understanding of the mechanisms and were accomplished by use of:

Scanning electron micrographs (SEM) and associated tungsten-mapping by energy dispersive analysis of X-rays (EDAX) (Figure 7);

Scanning electron detector (SED) micrographs and scanning Auger micrographs (SAM) which additionally map carbon, oxygen, and other light elements as well as tungsten (Figure 8);

X-Ray diffraction that shows the varying content of W_2C , $\beta\text{-WC}_{1-x}$ and WC on quenched burns resulting from different amounts of energy deposition (Figure 9);



(a) SEM



(b) W-distribution

Fig. 7. (a) Scanning electron micrograph and (b) an EDAX W-map of the area near a laser burn on WC/C. Note the liquid matrix phase, the attack on the side and end of one carbon fiber, and the growth of carbon on the end of a cooler nearby fiber.

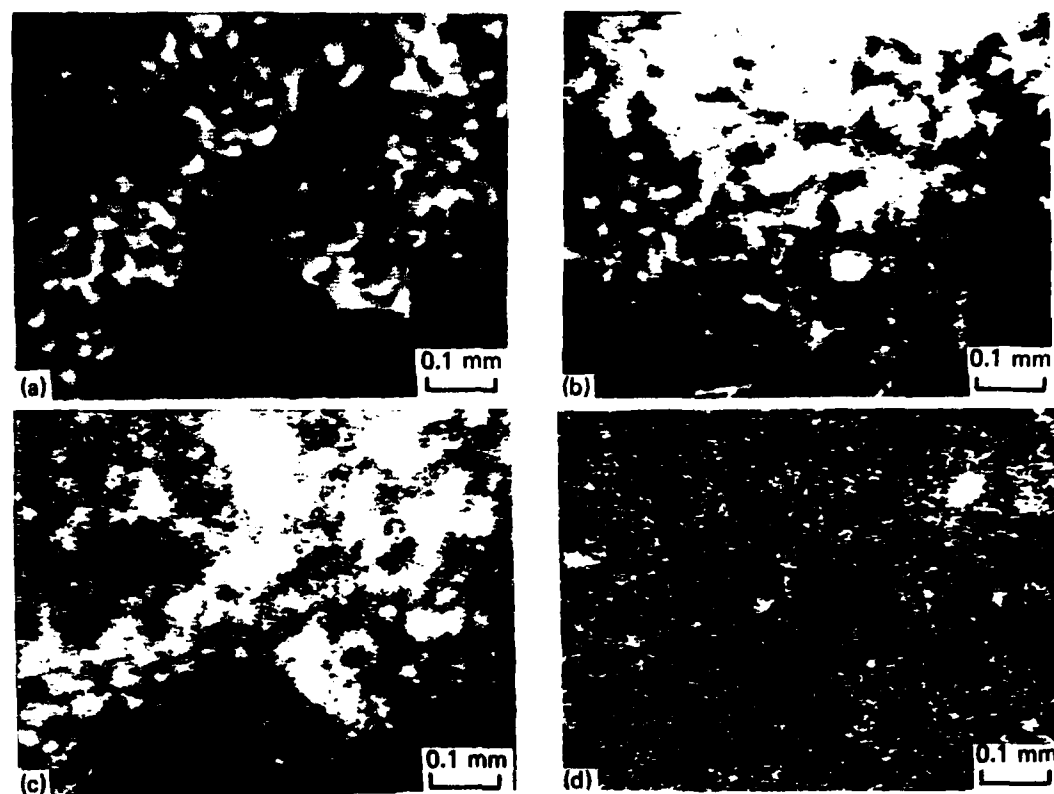


Fig. 8. (a) SED-produced micrograph of the edge of a laser burn on WC/C and SAM-produced (b) C-map, (c) O-map, and (d) W-map of the same area.

LASER-HEATED MATERIAL

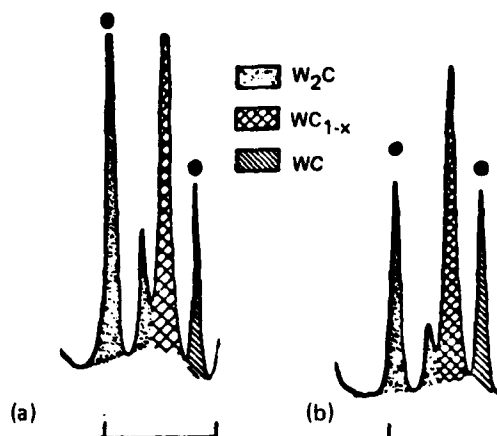


Fig. 9. X-Ray diffraction patterns from burn surfaces produced by different intensities of a CO_2 -laser beam interacting with WC/C. (a) 15 kW/cm^2 , (b) 30 kW/cm^2 . The higher energy flux intensifies the reaction of W_2C with carbon cloth to produce WC.

Optical photomicroscopy from metallographically prepared samples (Figure 10);

X-Ray radiography that reveals the movement of molten metal carbides within the burn craters (Figure 11) and can be used to compare the relative amounts and distributions of W in the various samples.

These tell a considerable amount about the course of mass movement as a result of laser-beam action. Most importantly the information gained verifies the liquifaction of the tungsten carbides, the changing composition of this liquid with increased radiant energy input, the segregation of the tungsten alloy at the edges of the burns, and the removal of carbon in the vapor state.



Fig. 10. Optical photomicrograph of the cross-section of a carbon wall formed downwind (to left) of a deeply penetrating laser-burn on WC/C in a 0.1 Mach wind tunnel.



Fig. 11. X-Ray radiograph of a laser-burned sample of WC/C showing ring of WC's pushed to the edge during radiation and droplets separating on the surface of the burn during cooling.

That the last named occurs is indicated by the spectra of C_2 and C_3 found immediately in front of the burn surface during laser irradiation, the large amount of carbon trapped in the burn surface when no burn-through occurs, and the redistribution of carbon downwind or in cooler parts of the carbon fiber composite. The wall downwind from the laser-produced cavity in Figure 10, for example, is pyrolytic in character (i.e., layered) which occurs when carbon is deposited from the vapor state.

The heavy ring of tungsten carbides about the burn in Figure 10 is indicative of mass flow during irradiation. The pressure exerted on the molten tungsten-carbon "alloy" due to gas evolution is partly the cause of the ring appearing. The cohesive character of this high melting fluid causes the thin residual film over the burn surface to break into droplets and also to add material to the ring.

The ring and the thin film remaining in the burn area frequently crack on cooling to relieve stresses but it is believed this arrests further shattering of the material that survives. It is also believed that thermal conduction through the cloth layers and through some modified matrix compositions further limits structural damage.

CORRELATION WITH OTHER EXPERIMENTS AND DATA

The final phase of this work involved the correlation of our work with other experiments and data. This entailed examination of the published literature, private communication with other scientists, and personal observations during experiments in other programs. This phase led to the use of the following:

Thermodynamic data¹ leads to equations (1), (2), and (3) which explain the high-energy absorption or "sink" that causes this material to survive a heavy energy flux.

The phase diagram of the tungsten-carbon alloy system² (Figure 12) shows the peritectic character of WC. On melting, the tungsten-carbon alloy becomes enriched in tungsten as it releases carbon, probably atom by atom.

The concept of jet cooling applied to some plasmas explains the fleeting disappearance of radiation due to dropping of radiation producing electrons to the ground level as vapor particles approach supersonic speeds in high vacuum³. Reradiation becomes possible as this supersonic kinetic energy is converted into internal electronic excitation with sudden deceleration to the velocity of one Mach. Figure 13 illustrates this effect.

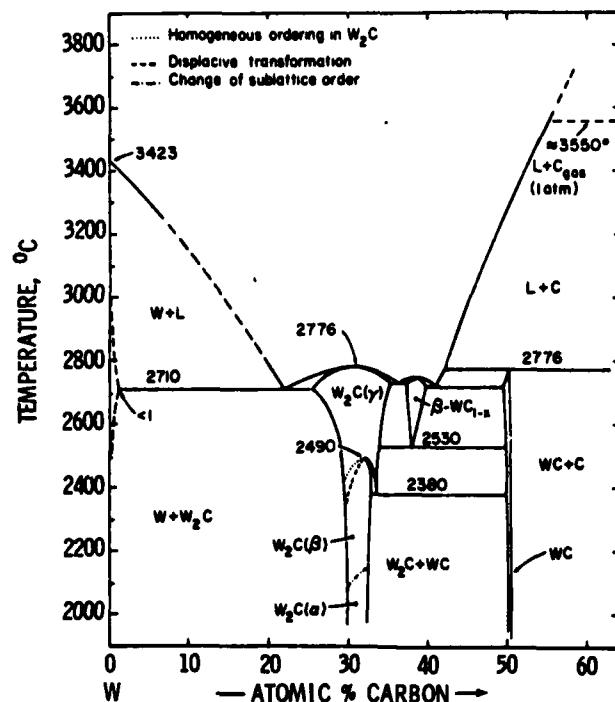


Fig. 12. Phase diagram of the tungsten-carbon system³.



Fig. 13. Free-jet expansion from vaporizing WC/C surface. Radiating C_2 and C_3 species close to the surface cool as supersonic speeds are reached, become invisible, and then reradiate strongly on deceleration to 1 Mach producing a "Mach-disk".

This final concept in the literature has let us infer the locale and significance of C_1 formation at high energy inputs. We propose that release of C_1 followed by formation of C_2 and C_3 is the dominant mechanism absorbing energy under such conditions.

Let us examine the structure of the radiant plume during CO_2 -laser radiation of our composite in vacuum. Directly in front of the irradiated area is a small bright plume that disappears farther from the surface and then blossoms into a large radiant "Mach disk" trailing into a diminishing tail "normal" to the irradiated surface, even though the angle of the impinging beam may fall at any angle up to 45° to the normal and produce the same effect. There is a thin barrel-shaped shock-wave connecting the perimeter of the burn area to the Mach disk not visible in this photograph. The disappearance of the original radiating plume is attributed to jet cooling.

We propose that the occurrence of the small radiating plume attached to the burn surface is related to release of C_1 from molten WC in this surface. This mode of vaporizing carbon absorbs the maximum amount of energy since six bonds connecting the graphitic carbon atoms must be severed instead of three or two of the C-C bonds to release C_2 or C_3 moities. If two C_1 's were to combine without escaping from the surface, the released energy of combination could produce one photon of UV-energy detectable only in the vacuum UV-region. However, if the combination energy is distributed between both electron activation, which can result in radiation, and kinetic energy as the new particles escape from the surface and approach supersonic speeds, the formation of C_2 and C_3 molecules can be occurring and produce this first bright plume. The new molecules could then produce the spectra observed in Figure 6.

Consequently, an examination of these plumes as near to the surface as possible in order to detect and measure any C_1 as well as the C_2 and C_3 already found there, would contribute to understanding the ablation mechanism and determine the relative importance of reflection and ablation for blocking the damage very high intensity lasers are capable of producing.

SUMMARY

In this paper we have attempted to show the means we have employed to reveal the nature of the interaction of a high-intensity infrared laser beam with a specific material system.

We have divided this study into four phases:

- (i) Initial material characterization
- (ii) "In situ" mass and energy changes during laser irradiation of the sample
- (iii) Post-radiation analyses of burns and surrounding areas
- (iv) Correlation with other experiments and data recorded in the literature, directly observed, or reported in private communications

which are illustrated by descriptions of the analytical techniques used and brief indications of the interpretations made of the acquired data.

We have presented an outline of the principal stages in the interaction of a particular laser (a cw 10.6- μ m CO₂-laser, welding type) with a particular composite material (a carbon cloth laminate with a pyrolysis-produced carbon and sintered mixed tungsten carbide matrix), and described a proposal for understanding the ablation mechanism involved.

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